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Standard Test Method for ~~Determining Aerobic Biodegradation in Soil of Plastic Materials or Residual Plastic Materials After Composting~~Determining Aerobic Biodegradation of Plastic Materials in Soil[1]

This standard is issued under the fixed designation D5988; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

~~1.1This test method covers determination of the degree and rate of aerobic biodegradation of synthetic plastic materials (including formulation additives that may be biodegradable) in contact with soil, or a mixture of soil and mature compost, under laboratory conditions.~~

~~1.2This test method is designed to rate the biodegradability of plastic materials relative to a standard in an aerobic environment.~~

~~1.3This test method is designed to be applicable to all plastic materials that are not inhibitory to the bacteria and fungi present in soil and compost.~~

~~1.4The values stated in SI units are to be regarded as the standard.~~

~~1.5~~

1.1 This test method covers determination under laboratory conditions of the degree and rate of aerobic biodegradation of plastic materials, including formulation additives, in contact with soil.

1.2 This test method is designed to measure the biodegradability of plastic materials relative to a reference material in an aerobic environment.

1.3 This test method is designed to be applicable to all plastic materials that are not inhibitory to the bacteria and fungi present in soil.

1.4 Claims of performance shall be limited to the numerical result obtained in the test and not be used for unqualified "biodegradable" claims. Reports shall clearly state the percentage of net gaseous carbon generation for both the test and reference samples at the completion of the test. Results shall not be extrapolated beyond the actual duration of the test.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific hazard statement is given in Section 8.

~~1.6This~~1.7 This ASTM test method is equivalent to ISO ~~17556:2003~~17556.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D425 Test Method for Centrifuge Moisture Equivalent of Soils
D618 Practice for Conditioning Plastics for Testing
D883 Terminology Relating to Plastics
D1193 Specification for Reagent Water
D1293 Test Methods for pH of Water
~~D1898Practice for Sampling of Plastics~~
D2980 Test Method for Volume Mass, Moisture-Holding Capacity, and Porocity of Saturated Peat Materials
D2989 Test Method for Acidity-Alkalinity of Halogenated Organic Solvents and Their Admixtures
D4129 Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 ~~on Environmentally Degradable Plastics.~~

~~Current edition approved November 1, 2003. Published January 2004. Originally approved in 1996. Last previous edition approved in 1996 as D5988-96. DOI: 10.1520/D5988-03.~~on Environmentally Degradable Plastics and Biobased Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

D4972 Test Method for pH of Soils
D5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures ~~D5511~~
D5511 Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions

2.2 *APHA-AWWA-WPCF Standards:*[3]
2540 D Total Suspended Solids Dried at 103°–105°C
2540 G Total, Fixed, and Volatile Solids in Solids and Semi-Solid Samples

2.3 *ISO Standard:*
~~ISO 17556:2003~~ISO 17556 Plastics—Determination of the Ultimate Aerobic Biodegradability of Plastic Materials in Soil by Measuring the Oxygen Demand in a Respirometer or the Amount of Carbon Dioxide Evolved

3. Terminology

3.1 *Definitions*—Definitions of terms applicable to this test method appear in Terminology D883.

4. Summary of Test Method

4.1 The test method described consists of the selection of plastic material ~~or compost containing residual plastic material after composting~~ for the determination of aerobic biodegradability, obtaining soil as a matrix and source of inoculum, exposing the ~~plastic materials or the compost containing residual~~ plastic material to the soil, measuring the carbon dioxide evolved by the microorganisms as a function of time, and assessing the degree of biodegradability.

4.2 The CO_2 production measured for a material, expressed as a fraction of the measured or calculated carbon content, is reported with respect to time, from which the degree of biodegradability is assessed.

~~4.3Alternatively,~~ 4.3 Alternatively, it is possible to determine the consumption of oxygen, or biochemical oxygen demand (BOD), ~~can be determined,~~ for example, by measuring the amount of oxygen required to maintain a constant gas volume in the respirometer flask, or by measuring the change in volume or pressure (or a combination of the two) either automatically or manually. The level of biodegradation expressed in percent is determined by comparing the BOD with the theoretical oxygen demand (ThOD). ~~The~~ In using this alternative approach, however, the influence of possible nitrification processes on the BOD ~~has to~~must be considered.

5. Significance and Use

~~5.1The degree and rate of aerobic biodegradability of a plastic material in the environment determines the extent to which and time period over which plastic may be mineralized. Disposal is becoming a major issue with the increasing use of plastics, and the results of this test method may permit an estimation of the degree of biodegradability and the time period over which plastics will remain in an aerobic soil environment. This test method determines the degree of aerobic biodegradation by measuring evolved carbon dioxide as a function of time that the plastic is exposed to soil.~~

~~5.2Soil is an extremely species-rich source of inoculum for evaluation of the biodegradability of plastics in the environment. When maintained appropriately with regard to moisture content and oxygen availability, the biological activity is quite considerable, although lower than other biologically active environments, such as activated sewage-sludge or compost. Soil is also the application target for composted materials, and therefore the biodegradability of such materials should be evaluated in the soil environment after the materials have been composted. A mixture of soil and mature compost containing composted plastic material (as obtained after performing Test Method D5338) is therefore also an appropriate matrix for evaluation of the biodegradability of plastics.~~

5.1 The degree and rate of aerobic biodegradability of a plastic material in the environment determines the extent to which and time period over which plastic materials are mineralized by soil microorganisms. Disposal is becoming a major issue with the increasing use of plastics, and the results of this test method permit an estimation of the degree of biodegradability and the time period over which plastics will remain in an aerobic soil environment. This test method determines the degree of aerobic biodegradation by measuring evolved carbon dioxide as a function of time that the plastic is exposed to soil.

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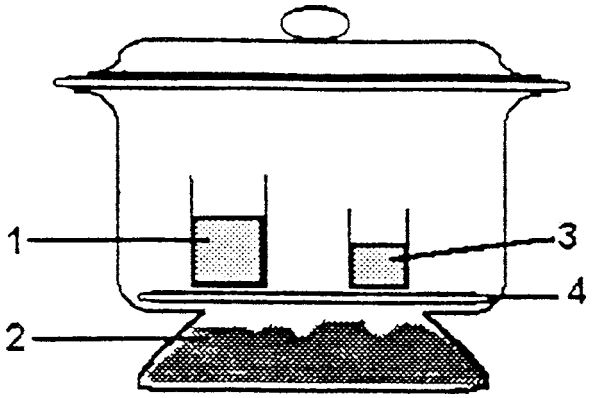
6. Apparatus

6.1 *Soil-Contact Incubation Apparatus* (see Fig. 1~~; biometer flasks are also appropriate):~~ ; biometer flasks are also appropriate). Ensure that all glassware is thoroughly cleaned and, in particular, free from organic or toxic matter.

6.1.1 *Vessels*~~, a set of approximately 2 to 4-L internal volume that can be sealed air-tight, such as 150-mm desiccators. For testing a plastic material in soil: three vessels for soil only controls, three for a positive control material, and three per test material.~~

[3] Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989, American Public Health Association (APHA), 1015 Fifteenth Street NW, Washington, DC 20005.



NOTE 1—(1) Barium hydroxide solution or potassium hydroxide solution, (2) soil, (3) water, and (4) perforated plate.

FIG. 1 Soil-Contact Incubation Apparatus

~~For testing a compost containing residual plastic material: three for soil only controls, three for a positive control material in soil, three for the compost-soil control, and three per compost containing test material (optional: three for the compost containing the positive reference from the previous composting test). In either case, three vessels may also be included as technical controls, containing only the absorbing solution and no soil.~~ , a set of vessels with approximately 2 to 4-L of internal volume with air-tight seal, such as 150-mm desiccators. Provide three vessels for soil only (known as "blanks" or "controls," these vessels show the background activity of the soil), three vessels for a positive reference material (these vessels show the viability of the soil microbial community), three vessels per test material, and three vessels as technical controls. The technical controls contain only the absorbing solution and no soil. The ambient air which fills the headspace of all the vessels introduces carbon dioxide into the system. The technical controls allow accounting for and subtracting this introduced carbon dioxide. Additionally, the technical controls indicate the air-tightness of the vessel system by showing possible infiltration of carbon dioxide into the sealed vessel.

6.1.2 *Beakers*, sets of 150-mL and 100-mL, equal in number to the soil incubation vessels.

6.1.3 *Perforated Plates or Other Support*~~, a set to hold the beakers above the soil inside each vessel.~~

~~6.1.4~~*, a set to hold the beakers above the soil inside each vessel. The support must be made from a material that will not absorb carbon dioxide.*

6.1.4 *Darkened Chamber or Cabinet*~~, in which the temperature is maintained at 21 ± 2°C.~~ , which allows selection of a temperature between 20°C to 28°C, and allows maintaining the selected temperature at ±2°C.

6.2 *Analytical Equipment*:

6.2.1 ~~*Analytical Equipment*~~Analytical Instrument, to measure the total carbon content of the test specimen.

6.2.2 *Analytical Balance*, to weigh the test specimen.

6.2.3 *Burette*, 100 mL.

6.2.4 *Bench-Top Centrifuge*, for moisture-holding capacity (MHC) determination.

6.2.5 *Oven*, set to 104 ± 1°C for moisture determinations.

6.2.6 *Muffle Furnace*, set to 550°C for ash determinations.

6.2.7 *pH Meter*.

~~6.3Alternatively, a flow-through apparatus or manometric apparatus as described in ISO 17566 may be used.~~

6.3 Alternatively, it is acceptable to use a flow-through apparatus or manometric apparatus as described in ISO 17566.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] ~~Other grades may be used,~~It is acceptable to use other grades, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 ~~*Ammonium phosphate*~~Ammonium Phosphate, $((NH_4)_2HPO_4)$, 4.72 g/L.

7.3 *Barium Hydroxide Solution* (0.025 N), prepared by dissolving 4.0 g anhydrous $Ba(OH)_2$/L of distilled water. Filter free of solid material, confirm normality by titration with standard acid, and store sealed as a clear solution to prevent absorption of CO_2 from the air. It is recommended that 5 to 20 L be prepared at a time when running a series of tests. When using $Ba(OH)_2$, however, care must be taken that a film of $BaCO_3$ does not form on the surface of the solution in the beaker, which would inhibit CO_2

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

diffusion into the absorbing medium. Alternatively, it is acceptable to use potassium hydroxide solution (KOH, 0.5 ~~N) could be used and is~~ N), prepared by dissolving 28 g of anhydrous KOH/L of distilled water and proceeding in the same way as for the ~~KOH.~~ $Ba(OH)_2$.

7.4 ~~*Hydrochloric acid*~~Hydrochloric Acid, 0.05 N HCl when using 0.025 N $Ba(OH)_2$, or 0.25 N HCl when using 0.5 N KOH.

8. Hazards

8.1 This test method includes the use of hazardous chemicals. Avoid contact with chemicals and follow the manufacturer's instructions and material safety data sheets.

9. Soil

~~9.1The soil can be a laboratory mixture of equal parts (by weight) of sand, topsoil, and composted manure or a natural soil sample. The soil should not be handled in any way that would inhibit the activity of the soil microorganisms. In the case of a natural soil, it is advisable to avoid soils that have been exposed to pollutants that may cause a significant perturbation of the microbial population. The source of the soil must be reported (see~~

9.1 Use natural, fertile soil collected from the surface layers of fields and forests. Make a laboratory mixture of equal parts (by weight) of soil samples obtained from at least three diverse locations (for example, an agricultural field, a forest, and a pasture or meadow). Taking soil from multiple and diverse locations will maximize biodiversity. It is advisable to avoid soils that have been exposed to pollutants that cause significant perturbations of the microbial population. The soils are preferably used fresh from the field to assure active microbiota. Air-dried or frozen soils must be reactivated before use in this test. It is preferable to use fertile soil classified as "sandy loam" in accordance with USDA classification, or "silty sand" in accordance with the German DIN classification.

9.2 The sources of the soils must be reported (see 14.1.1~~). The test soil may also be a mixture of a natural soil and a mature compost, as obtained at the end of Test Method D5338. A ratio of 1 g compost to 25 g soil corresponds to a typical application of approximately 120 tons of compost per hectare of agricultural land, assuming 20 cm of soil depth and a bulk density of 1.5 Mg m^{-3}~~.

~~9.2The soil is sieved to less than 2-mm particle size, and obvious plant material, stones, or other inert materials should be removed. The soil is then stored in a sealed container at 4 ± 1°C for a maximum of one month.~~

~~9.3The soil is analyzed for MHC by Test Method~~). Record the sampling site, its location, the presence of plants or crops, the sampling date, the sampling depth, and, if possible, the history such as details of fertilizer and pesticide application.

9.3 Sieve the soil to less than 2-mm particle size, and remove obvious plant material, stones, or other inert materials. Store the soil in a sealed container at 4 ± 1°C for a maximum of one month.

9.4 Analyze the soil for MHC by Test Method D425, Test Method D2980, or another analogous test method for MHC or field capacity.

~~9.4The~~ 9.5 Determine the pH of the soil ~~is determined~~ on a 5:1 (distilled water:soil) slurry using a glass combination electrode calibrated with standard buffers, following the guidelines given in Test ~~Methods~~Method D1293~~. The pH must fall between 6.0 and 8.0. (Soil with a pH above 8.0 may retain more of the CO_2 evolved by the microorganisms than a neutral soil, and soil with a pH below 6.0 may have an atypical microbial population.) Alternatively, the soil pH may be determined by Test Method~~ . Alternatively, it is acceptable to determine the soil pH by Test Method D4972~~.~~

~~9.5The moisture (total solids—dry solids) and ash (total solids—volatile solids) contents of the soil are determined in accordance with APHA-AWWA-WPCF 2540 D and G, respectively.~~ . The pH must fall between 6.0 and 8.0. (Soil with a pH above 8.0 retains more of the CO_2 evolved by the microorganisms than a neutral soil, while a soil with a pH below 6.0 has the potential to contain an atypical microbial population.)

9.6 Determine the moisture (total solids—dry solids) and ash (total solids—volatile solids) contents of the soil in accordance with APHA-AWWA-WPCF 2540 D and G, respectively.

9.7 It is acceptable for the test matrix to be a mixture of natural soil, as described in 9.1, and mature compost, such as obtained at the end of Test Method D5338. At a ratio of 1 g compost to 25 g soil, which corresponds to a typical application in agriculture of approximately 120 tons of compost per hectare of agricultural land (assuming 20 cm of soil depth and a bulk density of 1.5 Mg m^{-3}).

10. Test Specimen

10.1 Test specimens ~~should~~shall be of known weight and have sufficient carbon content to yield enough carbon dioxide that can be measured accurately by the trapping procedure described in this test method (see ~~11.1~~11.7 and ~~11.4~~11.9). ~~The~~ Determine the carbon content of the test material ~~may be determined~~ by calculation or elemental analysis, ~~according to~~in accordance with Test Method D4129.

~~10.2Test~~ 10.2 It is acceptable for test specimens ~~may~~to be in the form of films, pieces, fragments, powders, or formed articles, or in aqueous solution, ~~and they should be~~ in accordance with Practice D618~~. Any test specimens in the form of powders should be characterized as to particle size distribution by sieve analysis.~~

~~10.3Test specimens can be added directly to the soil matrix or, alternatively, after being submitted to a composting test (Test Method D5338). In the latter case, a homogenous and representative sample of the compost containing the residual plastics is used.~~